DEPARTMENT OF THE ARMY
COLD REGIONS RESEARCH AND ENGINEERING LABORATORY, CORPS OF ENGINEERS
HANOVER, NEW HAMPSHIRE 03755-1290

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Applied Research Branch

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Dear Charles:

The latest (and last) progress report from Dr. Jellinek is enclosed. He is now writing the final report.

Sincerely,

David Minsk

L. David Minsk
Research Physical Scientist
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PROGRESS REPORT IX
(March - June 1985)

De-icing and Prevention of Ice Formation of/on Offshore Oil-Drilling Platforms

Grantor: U.S. Army Corps of Engineers, CRREL
Hanover, New Hampshire 03755

Grantee: Clarkson University
Potsdam, New York 13676

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DATE SUBMITTED
7/85

Progress Report IX

(March - June 1985)

De-icing and Prevention of Ice Formation of/on Offshore Oil-Drilling Platforms

by

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Department of Chemistry

Clarkson University

Potsdam, NY 13676

Work on the most promising de-icing substances has been continued during this last period of the project.

(A) Dow Corning 3145 RTV Clear Adhesive Sealant.

This material proved very promising for de-icing.

(a) Adhesive Shear Strength.

The adhesive strength of the coats on an Al-substrate to ice was measured as a function of time. RTV-silicone and RTV-silicone mixed with Thomas silicone oil were tested. The results are listed in tables 1a and b, including some data presented in the previous report.

Table 1a
Samples of RTV and RTV Plus Thomas Oil Coats
Coated on Al-Substrate

Sample No.	RTV silicone paste/Thomas silicone oil, by wt.	Coater "clearance", mm
(a) 27.5% by vol. RTV silicone paste solution in toluene		
1	10/0	0.8
2	10/0	0.6
3	10/0	0.6
4	10/0	0.6
5	10/0	0.4
6	10/1	0.4
7	10/2	0.4
8	10/4	0.4
9	10/6	0.4
10	10/8	0.4
11	10/10	0.4
(b) RTV silicone waste (100% solid)		
12	10/0	0.4
13	10/0	0.6
14	10/0	0.8
15	10/0	1.5

Table 1b
Ice Shear Adhesive Strength of Samples Listed in Table 1a (-10°C)

Elapsed time after coating; days	Shear Adhesive Strength, $^{kg}/_{cm^2}$														
	Sample														
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1		1.53			1.03			0.57	0.47	0.28	0.30	0.26 (Gx3)			0.88
2			1.89									1.79	0.32	0.26	
3		1.11	1.07	1.08				0.24	0.28	0.11	0.19				
10	1.00					0.86	0.67 (Gx3)								
11	0.60					0.57	0.99								
12	0.56					0.79	0.88					2.01	1.14	1.06	0.51
13					0.61 (Gx16)							2.08	1.07	0.60	
14	0.60	0.40	0.40	0.51		0.64	0.66					1.35	0.77 (Gx3)	1.02 (Gx16)	
15												1.16	1.33		
16												1.19	1.36	1.15	
24							0.76 (Gx16)								
25							2.87								
82	3.73					3.52	4.08								
85					8.60			2.14	0.97 (Gx16)	0.97	0.64				
86									1.32		0.34				
87									0.90		0.28	3.11	2.74	2.15	
97															
98									0.29	0.11	0.12				

Note: e.g., "Gx16" means "applied water jet for 16 hrs.".

Some remarks are made below concerning the results in Table 1b:

(1) Table 1b shows that coats without silicone oil (samples Nos. 1 to 5 and 12 to 15) have smaller adhesive strengths with elapsed time than those with silicone oil. The decrease reaches a minimum value within two to three weeks. Subsequently, the values increase to a high value which is beyong that specified by the sponsor (i.e. 1.76 kg/cm^2).

(2) A dependence of the adhesive strength on coat thickness was not found.

(3) Coats prepared from solution exhibited a number of tiny irregularities on their surface; their shear adhesive strength was somewhat larger than that of coats made from paste. Thus it was thought desirable to carry out tests using a mixed solvent system, so that smooth vaporization could be achieved once the solution was sprayed on the Al-substrate.

(4) Coats containing at least 60% by wt. of silicone oil (Nos. 6 to 11) had low adhesive strengths for at least 98 days; it is expected this would last for a considerably longer time.

(5) The water erosion affected the adhesive strength of the coats; but the values remained within the limits of the specified value for coats containing 60% by wt. of silicone oil.

II. Tensile Strength of RTV Silicone Coats.

Films (coats) prepared from paste were put on various substrates from which they could be easily removed as films. The tensile strength of these films was measured after curing at room temperature for 1, 2, 3 or 5 days. Films of different thickness were obtained by using coaters of different gap-widths. Table 2 gives remarks.

Table 2
Tensile Strength of RTV Films
Removed From Their Substrates
(Prepared from paste; room temperature)

Elapsed time after coating, days	Tensile modulus, kg/cm^2			
	Film A: Thickness			
	263 ± 8 μm	398 ± 8 μm	578 ± 11 μm	Av. kg/cm^2
1	6.81	7.00	7.38	7.06
2	8.53	7.42	8.84	8.26
3	5.47	7.92	7.99	7.13
6	5.17	6.14	8.12	6.48
7	6.35	6.70	9.66	7.57
Ave. (kg/cm^2)	6.47 ± 1.3	7.04 ± 0.7	8.40 ± 0.9	7.30 ± 0.7
	Film B: Thickness			
	229 ± 6 μm	382 ± 6 μm	529 ± 6 μm	Av. kg/cm^2
1	7.97	6.70	8.78	7.82
2	6.79	8.95	8.84	8.19
3	7.22	7.83	7.82	7.62
6	5.45	6.89	9.22	7.19
7	7.80	8.40	9.75	8.65
Ave. (kg/cm^2)	7.05 ± 1.0	7.75 ± 1.0	8.88 + 0.7	7.89 ± 0.5

Note: Film A was prepared by coating RTV silicone paste on a glass plate and Film B on polypropylene film, respectively. The former coat could be removed as film by using a razor blade and the latter was easily removed by peeling; the latter film was less damaged.

The average strengths in table 2 increase systematically with film thickness, while the strengths as a function of time do not vary in an orderly way. This indicates that the main effect is due to film thickness while the elapsed time plays a minor role. Sample No. 12 in table 1a and b (295 µm thick) was measured after 98 days had elapsed; its tensible strength was 8.8 kg/cm^2. This value is larger that the average value of 7.1 ± 1.0 kg/cm^2 (table 2, film b; av. thickness 229 µm). The value is also larger than the av. value for film a, (6.87 ± 1.3 kg/cm^2; 263 µm). The elapsed times giving these average values were much shorter than 98 days (i.e. 3.5 days) for sample 1b. A long elapsed time increases the tensible strength. This is due to continued crosslinking taking place in the film (coat). That RTV is still crosslinking after such long time was shown by us using I.R. spectra. Optical densities of O-H bonds (2.9 µm) changing with time were taken as indicators for this crosslinking process.

Final Conclusions

Four coatings suitable for de-icing of oil-drilling platforms have been found whose shear adhesive strength (-10°C) lies well within the specified adhesive strength (1.76 kg/cm^2) after "erosion" by a water jet.

(1) Poly(dimethyl siloxane)-bis phenol-A-polycarbonate block co-polymer LR5630 (G.E. Co.) with silicone oil SF-1154 (G.E. Co.).

The composition of the "spraying" solution is as follows:

Co-polymer LR5630 10 g.; SF-1154 5 g.; toluene 40 ml. This solution is prepared by dissolving LR5630 in toluene under stirring and then adding SF-1154 silicone oil. The thickness of the coat was ca. 0.4 mm i.e. 25 ft^2/gal. would give such a thickness.

Shear adhesive strength (-10°C) before erosion <0.07 kg/cm^2.

Shear adhesive strength (-10°C) after erosion (16 h) <0.61 kg/cm^2.

(2) Crosslinked PE (polyethylene form), Nalgene 6281 series, thickness 3 mm.

The top surface is coated with "silicone masonry sealer". A "pressure sensitive adhesive" is applied to the bottom surface of the foam, this bottom surface is adhered to the substrate.

Shear adhesive strength before erosion (-10°) 0.24 kg/cm^2.

Shear adhesive strength after 16 h erosion (-10°C) 0.20 kg/cm^2.

Shear adhesive strength additional 16 h 0.28 kg/cm^2.

The top surface can also be covered with "masonry sealer" for dust-protection (True Value).

(3) Dow Corning 3145 RTV clear adhesive sealant with Thomas silicone oil.

Thickness of coat ca. 0.1 mm.

Solution: 3145 RTV 27.5% by vol. is dissolved in toluene under stirring (room temperature), subsequently silicone oil is added.

Ratio by wt. 3145 RTV/Thomas oil 10/6.

Shear adhesive strength before erosion (10°C) 0.97 kg/cm^2.

Shear adesive strength after 16 h erosion (-10°C) 1.32 kg/cm^2.

Shear adesive strength one day after erosion (-10°C) 0.90 kg/cm^2.

Thickness of coat ca. 0.1 mm, i.e. 180 ft^2/gal.

(4) Dow Corning varnish #997.

Curing conditions 200°C, 4.5 h.

Shear adhesive strength before erosion (-10°C) 1.45 kg/cm^2.

Shear adhesive strength 16 h erosion (-10°C) 1.43 kg/cm^2.

Thickness of coat ca. 0.2 mm, i.e. 100 ft^2/gal.

De-icer (1) is the most efficient one while de-icer (2) is easiest in application.